EVALUATION OF Ni – ALUMINA COMPOSITE ELECTROPLATING ON MILD STEEL

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Report submitted in partial fulfillment of the requirements
For the award of the degree of
Bachelor of Mechanical Engineering

Faculty of Mechanical Engineering
UNIVERSITI MALAYSIA PAHANG

NOVEMBER 2010

SUPERVISOR'S DECLARATION

I hereby declare that I have checked this project and in my opinion, this project is adequate in terms of scope and quality for the award of the degree of Bachelor of Mechanical Engineering.

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STUDENT'S DECLARATION

I hereby declare that the work in this thesis is my own except for quotations and summaries which have been duly acknowledged. The thesis has not been accepted for any degree and is not concurrently submitted for award of other degree.

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ACKNOWLEDGEMENTS

First I would like to express my grateful to ALLAH S.W.T. as for the blessing given that I can finish my project.

First, I have engaged with many people in helping me completing this project. I wish to express my sincere appreciation to my main thesis supervisor Mdm Dayangku Noorfazidah Awng Shri, for his germinal ideas, continuous encouragement, invaluable guidance, advices and motivation. Without his continued support and interest, this thesis would not have been the same as presented here.

The next category people who help me to grow further and influence my project are my previous panels, Dr Sugeng Ariyono, Mr. Mohd Firdaus Hassan, Mr. Nasrul Azuan Alang that advice me on how to complete this project greatly. I would like to acknowledge their comments and suggestions, which was crucial for the successful completion of this study. Also to my colleagues who always help me in order to finish this project. I would like to express my gratitude especially to all FKM laboratory instructors and for them help and advices. I appreciate very much to them because of their excellent co-operation, inspirations, idea and support information given during this study.

Last but not least I acknowledge without endless love and relentless support from my family, I would not have been here. My father, Jamil bin Ajang, my mother, Zalinah Bte Mohd Zin, all my sisters and brother that always support, sacrifice, patience, understanding that were inevitable to make this work possible, motivation and encourage me to success. I cannot find the appropriate words that could properly describe my appreciation for their devotion, support and faith in my ability to attain my goals.

ABSTRACT

This thesis deals with an evaluation of Ni- Alumina composite electroplating on mild steel. Electroplating process has been carried out by utilizing the Watt's Bath with Alumina oxides powder. Weight changes in both electrode shows that weight loss of anode and weight of deposit on cathode are proportional to plating time and voltage supplied. There are three parameters involve in this project that are Alumina concentration, plating time, and voltage supplied. Mild steel will be the anode and connected to positive terminal. Meanwhile, nickel plate as anode will be connected to negative terminal. Dimension of sample in this project is 80 mm x 30 mm x 1.5 mm. Nickel ion is reduced to form or plate a surface on mild steel. The electroplating bath contains the metal ion to be reduce as well as Alumina oxides to support the flow of electrons. While reduction of the metal occurs at cathode, oxidation is simultaneously occurring at anode. After that, microstructural analysis was done to observe the thickness of coating and its surface structure. Lastly, all the samples were undergoes Vickers hardness test. In this project, it can be conclude that result shows increase of Alumina concentration, plating time, and voltage supplied also increase the thickness of coating.

ABSTRAK

Tesis ini berkaitan dengan penilaian penyaduran Ni-Alumina komposit pada besi tulen. Proses elektroplating dilakukan dengan mengaplikasikan Mandi Watt dengan serbuk oksida Alumina. pPrubahan berat dalam kedua-dua elektrod menunjukkan bahawa kehilangan berat di anod dan berat bertambah di katod setanding dengan tempoh penyaduran dan voltan disediakan. Ada tiga parameter yang terlibat dalam projek iaitu kepekatan Alumina, tempoh sadur, dan voltan disediakan. baja ringan akan menjadi anod dan dihubungkan ke terminal positif. Sementara itu, plat nikel sebagai anoda akan disambungkan ke terminal negatif. Dimensi sampel dalam projek ini adalah 80 mm x 30 mm x 1.5 mm. Ion nikel berkurang untuk membentuk atau plat permukaan pada besi tulen. Cecair penyaduran mengandungi ion logam dapat mengurangkan serta Alumina oksida untuk menyokong aliran elektron. Sementara pengurangan logam terjadi pada katod, pengoksidaan secara serentak terjadi pada anod. Setelah itu, analisa mikrostruktur dilakukan untuk melihat ketebalan lapisan dan struktur permukaannya. Akhir sekali, semua sampel mengalami uji kekerasan Vickers. Dalam projek ini, dapat disimpulkan hasil tersebut menunjukkan peningkatan kepekatan Alumina, tempoh sadur, dan voltan yang disediakan juga meningkatkan ketebalan lapisan.

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LIST OF SYMBOLS

-	Negative
+	Positive
μ	Micron
%	Percentage
°	Angle / Degree
θ	Degree

LIST OF ABBREVIATIONS

AISI	American Iron and Steel Institute
ASTM	American Society for Testing And Material
SEM	Scanning Electron Microscope
UNS	Unified Numbering System
PSM	Projek Sarjana Muda
PVD	Physical Vapor Deposition
SHS	Self-Propagating High Temperature Synthesis
CED	Conventional Electro Deposition
et al	And Others

CHAPTER 1

INTRODUCTION

1.1 PROJECT BACKGROUND

Electroplating is a method of covering objects with a thin layer of metal. Discovered by Micheal Faraday in the 1830's it has contributes to development and application in many areas of industry besides our daily lives in many ways.

In this project, the electroplating process has been carried out to observe the dissolution of cast nickel anode together with alumina powder and the deposition of metallic nickel alumina on the mild steel cathode. Besides, this experiment will establish the effect of various electroplating parameters of nickel alumina deposition.

During electroplating, the Nickel Sulphate deposited into Ni^{2+} ion and SO_4^{2-} ion according to this chemical reaction:

$$NiSO_4^{2+} \rightarrow Ni^{2+} + SO_4^{2-} \tag{1.1}$$

While the water will also dissociates into:

$$H_2\text{O} \rightarrow H^+ \ + \ OH^- \tag{1.2}$$

The numbers of positive and negative ion charges exists equally in the solution. The object to be plated (cathode) is connected to the negative (-) side of the battery, giving it the negative charge, and the plating metal (anode) is connected to the positive (+) side of the battery, giving it a positive charge. Positive ions flow the anode toward the object being plated, through the plating solution (electrolyte), and finally deposited onto the surface of the object.

In this experiment, since the anode is the same metal of the solution, the anode will deposited to form Ni^{2+} ions and form a positively charged outer under applied condition. On the other hand, at the cathode, its outer layer performs a negatively charged layer under the same applied condition after receiving electron from the current supply. Furthermore, this will attracts Ni^{2+} ions in the solution then replaced by the Ni^{2+} ions from the Nickel anode. So, the concentration of Ni^{2+} ions in the solution is remains constant till the anode fully deposited.

1.2 PROJECT OBJECTIVES

In order to accomplish the objectives of performing the experiment, four electroplating parameters: voltage, plating time and Alumina concentration, had been vary-related into experiments.

a) To determine the effect of various relations of voltage and plating time to the weight of nickel deposit in a constant temperature condition.
b) To determine the effect of different concentration of Alumina to the weight of nickel deposit in constant voltage and plating time.
c) To determine the coating properties such as the hardness and its microstructure by undergoes coating analysis.

1.3 SCOPE OF PROJECT

a. In order to accomplish the set objectives, the following scope of works has been drawn:
b. To perform the electroplating process of Ni-Alumina composite on mild steel at constant and difference voltage.
c. To perform the electroplating process of Ni-Alumina composite on mild steel at constant and difference plating time.

d. To perform the electroplating process of Ni-Alumina composite on mild steel at constant and difference concentration of Alumina.
e. Microstructure analysis on coating.
f. To perform Vickers Hardness Test on coating.

1.4 REPORT ORGANISATION

Based on the project process flow chart, the experiments were started with identify the problems and objectives which related to my title given: Evaluation of Ni-Alumina Composite Electroplating on Mild Steel. Besides, all information was gathered from journals, books, magazines, and websites to finish literature review's part.

When it comes to methodology part, journal has been used as main reference. All information from journal was gathered and understands by discussing with supervisor and technical staff to complete it one by one step procedure. So, analysis parameters were confirmed. They are alumina concentration, time of plating, and plating voltage. The parameters will be various at every different experiment. After that, the analysis of coating test was confirmed, which are its thickness, microstructure observation, and Vickers test. The microstructure observation was done by using inverted microscope and optical microscope and last but not least, hardness Vickers tester was used to undergoes hardness test on coating.

Then, all equipment was prepared. This includes all hardware such as beaker, mild steel and nickel plate besides chemicals to make electroplating solution. Experiment was setting up after done with its preparation. Each of material was confirmed available to be use. The final analysis of experiment was done to make sure everything under good conditions. It will be proceed if the analysis result were okay and if it's not, then it will go back again to start level and re-do from the beginning.

The data from the experiment were collected. This includes the result of plating at before and after conditions. The collected data were structured in a table. Discussions were made after experiment and analysis on coating done successfully. After that, conclusion will be made together with some recommendations.

Last but not least, it will be project presentation. All the material and data were gathered to do slide presentation and finish the report. It follows by submission of report.

1.5 PROJECT FLOW DIAGRAM

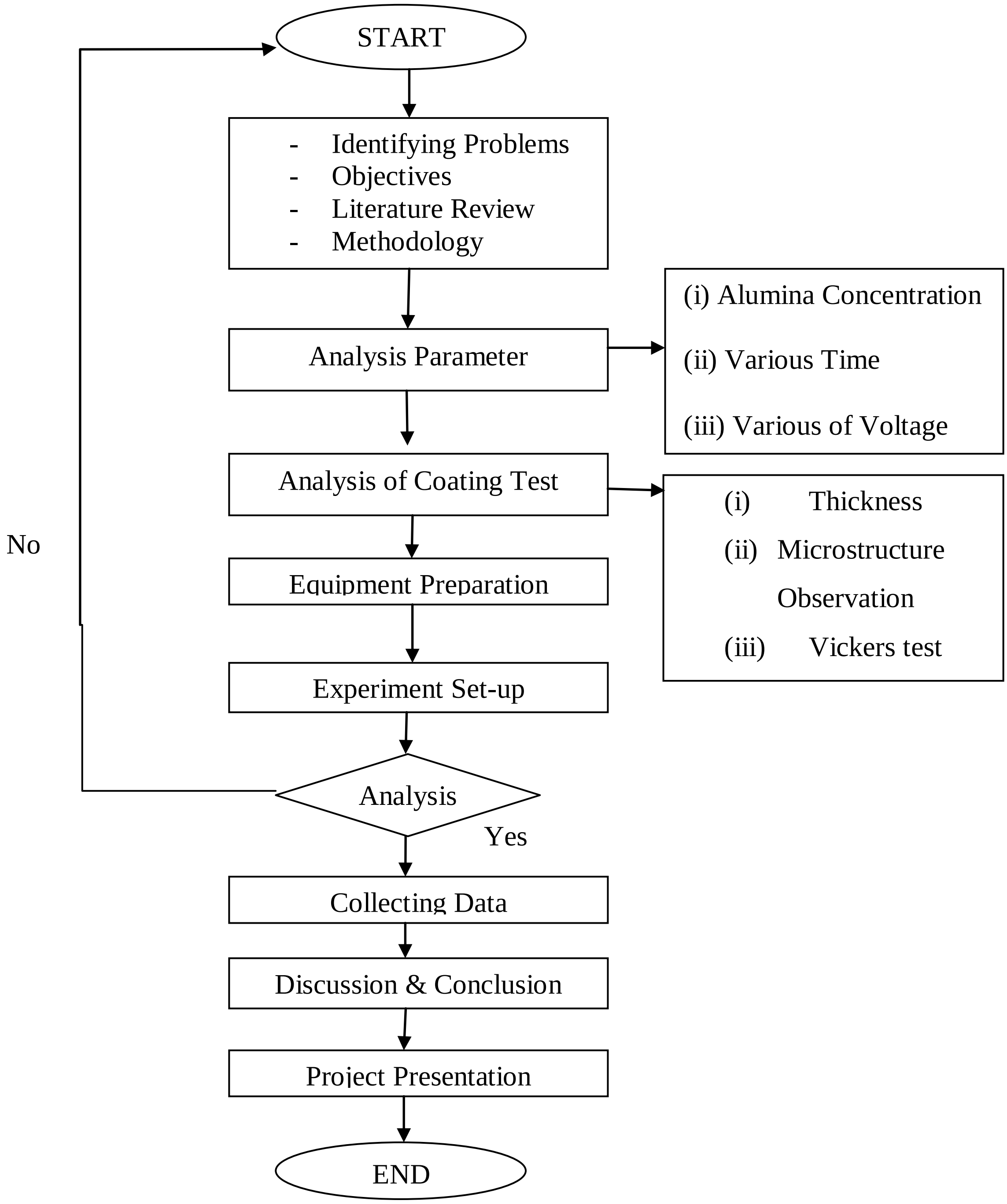

Figure 1.1: Project process flow chart

CHAPTER 2

LITERATURE REVIEW

2.1 INTRODUCTION

Electroplating is a plating process that uses electrical current to reduce cations of a desired material from a solution and coat a conductive object with a thin layer of a material, such as a metal. It is a multidiscipline of engineering, mechanical, and electrical, in co-ordination with applied chemistry. Present day electroplating has become a well-established branch of metal finishing.

Adding ceramics and intermetallics into regular metal coatings is becoming a common method to improve surface properties. Electro-deposition is well established method for fabrication of such materials because of advantages in uniform depositions on complexly shaped substrates, low cost, good reproducibility and the reduction of waste.

2.2 HISTORY Of ELECTROPLATING

2.2.1 Samples of Previous Electroplating Research

The development of electroplating has been described in detail by Hunt (1973), Kramer Weiner and Fett (1959) and Pavlova (1963). An aqueous solution of metal salts and a source of electricity are the chief requirements for electroplating, so the early efforts were concentrated on the search for a good electrical source (Wiley,1963).

The commonly accepted opinion is that the first man to deposit metal from its chemical compounds was Professor G.B. Beccaria in 1722, back in the days of the phlogiston theory (phlogiston being something which was thought to be needed to generate metals). The energy, or electricity, was produced by a Layden jar, which acted as a capacitor, and provided a means of storing electricity, but could only supply weak, short pulses of current (Arthur Kenneth Graham, 1971)

In 1971, Luigi Galvani of the old and famous university of Bologna discovered that muscle tissue of frog reacts to pulses of electric current. He observed that a frog's muscle suspended on a copper ring convulses on contact with iron. The matter generating these convulsions was named fluidum and mistakenly thought to be a property of the animal tissue itself, but identical with electricity. The effect was named galvanismus after Galvani. The story of the Elkington cousins, George Richards and Henry Elkington, is very complicated and even L.B. Hunt had problems unraveling it. G.R Elkington referred to himself as a 'Gilt Toy Maker'; he and Henry made small articles like military badges, buttons, and snuff boxes. He was interested in replacing the dangerous amalgamation process for gilding with something less poisonous and easier to handle. The earlier patents of the Elkington mostly covered immersion gilding processes but on March 25th, 1840 the cousins filed a patent (B.P. 8447) 'Improvements in Metals', detailing silver and gold solutions in connection with the application of current. The source of current was very important:' …a solution of chloride of sodium…into this a cylinder of zinc is immersed, with a wire of copper…', clearly it is an electrochemical battery. This patent claim gave Werner von Siemens an opening. He sent his younger brother Wilhelm to England to negotiate his own patent claims with the Elkingtons. This ultimately led to Wilhelm becoming famous as Sir William Siemens, industrialist and scientist, and honored with a memorial window in Westminster Cathedral (Nasser Kanani, 2004).

The work of the scientist Michael Faraday in the 1830s, in generating electricity by elelctromagnetic dynamo was also initially unappreciated commercially. At a meeting in 1850 of the British association, the Elkington cousins still expressed a preference for batteries over the dynamo, even though they had the patents for dynamo since 1846. However, their statements were possibly mere polemic as large Magneto macj=hines were installed at that time in their Newhall Street, Birmingham works, with a capacity to deposit up to 50 ounces of silver in an hour (Mogerman,1974).

Alloy plating where two (or more) metals are simultaneously deposited is increasingly used for decorative finishes. For instance steel can be plated with brass by using an electrolyte of copper and zinc salts in the ratio 60:40 dissolved in a solution of sodium cyanide. Similarly other alloys such as bronze, speculum, lead-tin, etc, can be produced as platings (H.Silman, 1978).

2.2.2 Process Of Ni-Alumina Electroplating

Electroplating is an electrochemical process by which metal is deposited on a substrate by passing a current through the bath. In the Ni- Alumina electroplating process, the anode, nickel plate is connected to the positive terminal of the power supply. On the other hand, the cathode, which mild steel is connected to the negative terminal. The anode or nickel plate will be the source of the material to be deposited and the cathode which is the substrate with negatively charged electrode is to be coated. Besides, the dissolution of cast nickel anode together with alumina powder will result the deposition of metallic nickel alumina on the mild steel cathode.

2.2.3 Nano-Composite Coating

Recent years, nano-composite coating has got much attention for its excellent mechanical properties such as wear resistance, corrosion resistance and lubricant. However, it has been not easy to elevate the content of nano-particles by traditional way up to now. It is well known that the properties of the composite coatings are heavily dependent upon the degree of particle incorporation in the deposit. Higher incorporation percentages and more uniform distribution of inert particles in the metal matrix lead to the improvement of the mechanical, tribological, anti-corrosion and anti-oxidation properties of coatings. In the last three decades, micro and nano-sized inorganic inert particles with metal or alloy such as SiC,ZrO_2, Al_2O_3, Si_3N_4, TiO_2, CeO_2, nano-diamond, carbon nanotubes (CTN), etc.

Previous experiment, effect of micro and nano sized particles such as SiC and Al_2O_3, on different material properties were investigated. Other ceramics like WC, CeO_2 and TiO_2 were successfully electrodeposited and examined too. Electro-deposition of nickel aluminum composite coatings were accomplished in Watts bath based of following chemistry: 300g/dm^3 $NiSO_4.7H_2O$, 45g/dm^3 $NiCl_2.6H_2O$, 40g/dm^3 H_3BO_3, with addition of 15 g/l $Al_2(SO4)_3$, 10 g/l $(NH_4)2SO_4$ and 40 g/l aluminum powder (mean diameter 12 µm). The pH of all solutions which measured set to 3.5 at room temperature. Deposition time was fixed at 30 min. Before plating, the substrate surface was mechanically polished with abrasive paper. The experiment were performed in a 5kW pulsed plasma processing cold wall unit. Three measurements were conducted on each sample and the results were averaged.

- Effect of current density on aluminum particle co-deposition

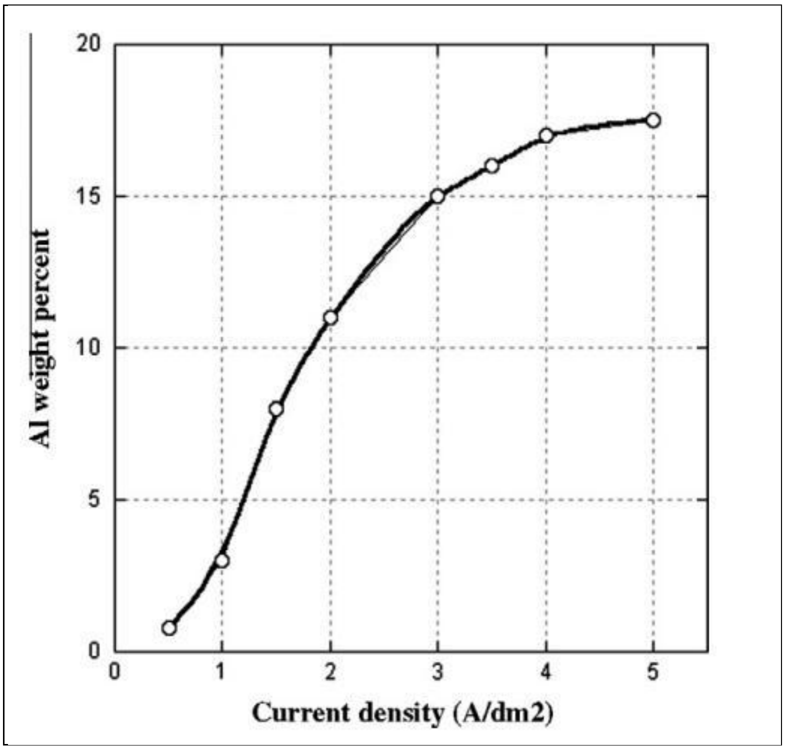

Figure2.1: The Al particle weight percent as a function of current density.

Source: Daemi et al (2010)

The structure and properties of composite coatings depend not only on the concentration, size, distribution, and nature of the reinforced particles, but also on the type of using solution and electroplating parameters such as current density, temperature, and pH value. Chemical composition of different electrodeposited Ni-Al composites, produced by different current densities was analyzed by use of X-ray fluorescence spectroscopy and the result shown in Figure 2.1.

Figure 2.1 shows that by increase of current density the incorporation of aluminum increase in the coating. It can be seen that the slope of the curve is high at current densities below $2A/dm^2$ and as the current density exceeds $3A/dm^2$, the slope decreases noticeably. Increase in Al particulates co-deposition can be attributed to the increasing tendency for adsorbed particles to arrive in the cathode surface

- Surface topography

SEM micrographs from the surface of the samples are illustrated in Figure 2.2.

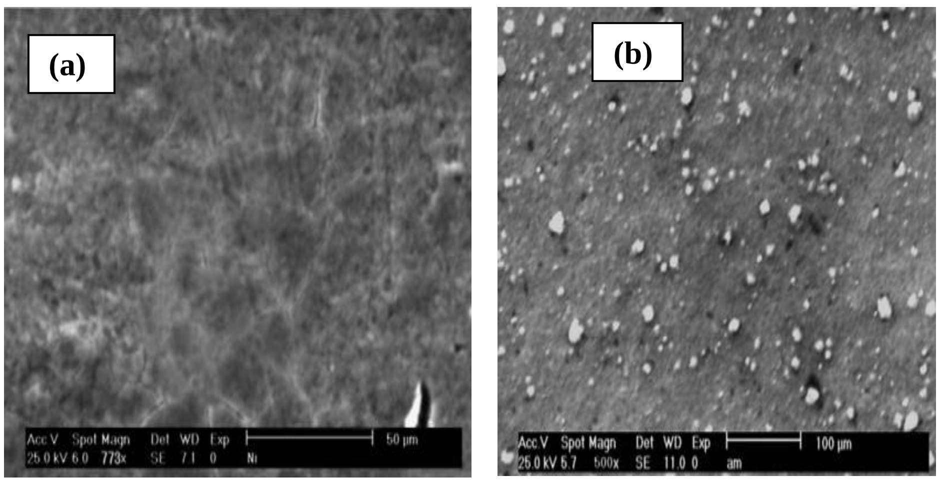

Figure 2.2: Surface morphology of composite coating

Source: Daemi et al (2010)

Figure 2.2 (a) shows pure Ni coating and figure 2.2 (b) shows Ni/Al composite coating. Comparison of Figure 2.2(a) with Figure 2.2(b) shows that the Al particles appear as the light spots in the darker nickel matrix. Besides, the size of particles is bigger than the matrix roughness and they stick out of nickel matrix. Furthermore, as can be seen, pure nickel coating has smoother surface than Ni-Al coating. By increasing embedded particles, the surface become rougher and the roughness of coating are about two and three times more than pure nickel coating.

- Microhardness measurement

Vickers microhardness (HV) values of the selected coatings are tabulated in Figure 2.3.

Sample	Coating	Microhardness ($HV_{0.015}$)	Roughness R_a (μm)
1	Pure Ni	280	0.17
2	Ni/Al (11 wt%)	275	0.36
3	Ni/Al (14 wt%)	270	0.45
4	Ni/Al (11 wt%) + PN	530	0.64
5	Ni/Al (14 wt%) + PN	620	0.73

Figure 2.3: The microhardness and surface roughness of different samples

Source: Daemi et al (2010)

Figure 2.3 shows clearly that incorporation of aluminum in nickel matrix decreases the microhardness of the coating to some extent. The mean value of Vickers microhardness of pure nickel coating has been found of about 280HV while that of composite coating is less than it. According to the rule of mixture the composite hardness can be estimated by Eq 2.1.

$$H = \frac{1}{\frac{fh}{Hh}+\frac{fs}{Hs}} \tag{2.1}$$

Which;

Hh = Hardness values of hard phases

Hs = Hardness values of soft phases

fh = volume fraction of hard phases

fs = volume fraction of soft phases